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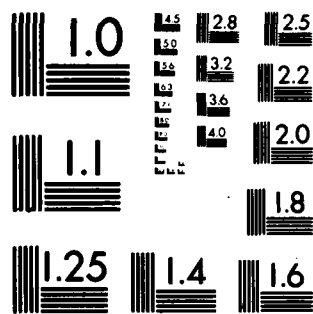
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TECHNICAL REPORT, NO. 21

TBA Studies of Prepreg Curing Behavior.

by

N. S. Schneider and J. K. Gillham

Prepared for Publication

in

Polymer Composites

Princeton University  
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Department of Chemical Engineering  
Princeton, NJ 08544

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times as a function of temperature, or in constant heating rate scans to reveal the  $T < T_g$  and the  $T_g$  relaxations of the uncured resins, and at higher temperatures phenomena associated with gelation, vitrification and devitrification.

## TBA STUDIES OF PREPREG CURING BEHAVIOR

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## ABSTRACT

→ The torsional braid analysis (TBA) equipment has been used as an automated torsion pendulum to characterize prepreg materials in the form of single ply strips ( $2\frac{1}{2} \times \frac{1}{8}$  inch). Compared to the use of coated glass braids, the main difference was a marked weakening of the gelation mechanical damping peak in isothermal runs. However, prepreg materials consisting of epoxy resins on glass, carbon or aramid fibers were successfully run isothermally to provide gelation and vitrification times as a function of temperature, or in constant heating rate scans to reveal the  $T < T_g$  and the  $T_g$  relaxations of the uncured resins, and at higher temperatures phenomena associated with gelation, vitrification and devitrification.

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## INTRODUCTION

Several studies have been reported on the curing behavior of thermosetting resins employing torsional braid analysis (TBA) with the samples coated on a glass fiber braid (1-3). During isothermal cure two mechanical loss peaks are usually observed as a function of increasing cure time. These peaks have been attributed to gelation and vitrification, respectively. Since the times to gel and to vitrify are key parameters which describe the cure behavior, they are useful in prepreg technology and composite processing. Temperature scans on the uncured resin reveal glassy state ( $T < T_g$ ) relaxations, the glass transition temperature ( $T_g$ ), and at successively higher temperatures gelation, vitrification and devitrification processes. Scans on the cured resin, on cooling from the maximum temperature of cure, by comparison, depict the change in  $T_g$ , rigidity, and low temperature ( $T < T_g$ ) damping relaxations which characterize the extent of cure.

Earlier results illustrating the behavior described above have been reported for two "250°F cure" resin systems (3). The purpose of the present study was to determine whether the same information could be obtained working directly with prepreg materials. Supporting results obtained by DSC will be referred to but the primary emphasis here is on the TBA data.

## EXPERIMENTAL

Three commercial unidirectional prepreg samples were studied: "Prepreg 1", one of the above "250°F cure" resins (3) which is based

on the diglycidyl ether of bisphenol A/epoxy cresol novolac, dicyandiamide curing agent and an accelerator, on glass fibers [source, 3M (SP250/S2)]; "Prepreg II", a "350°F cure" resin consisting of tetraglycidyl methylenedianiline, with diaminodiphenylsulfone as curing agent, on Kevlar<sup>(R)</sup> fibers [source, Fiberite (HYE-1734A, 934/Kevlar); and "Prepreg III", a second "350°F cure" resin, presumably similar to the preceding resin, on carbon fibers [source, Hercules (3501-6 AS)]. Samples were stored in a freezer until required.

All TBA measurements were carried out on an automated system under a helium atmosphere at a nominal frequency of 1 Hz (1,2). DSC scans were conducted using the Perkin-Elmer DSC II equipped with a two-stage intercooler and purged with nitrogen. Runs were made on samples of about 10 mg at 5°C/min for curing studies and at 20°C/min to determine the  $T_g$  of uncured resin.

#### Effects of Sample Geometry

Prepreg strips, 2-1/2" in length and approximately 1/8" in width were mounted in simple, flat faced, screw-tightened grips. The results obtained for I cured isothermally at 90°C (Figure 1) show a gel peak in the damping curve which is very much weaker than that obtained when glass braid is used as a support (1-3), but the peak is readily identifiable. The vitrification peak remains strong. The reduction in the intensity of the gel peak for the prepreg is due, presumably, to the fact that the prepreg does not act as a coherent structure in torsion since the liquid resin is the only means of coupling between the fibers.



As a result apparently very little shear is imparted to the resin until curing proceeds past the gel point and establishes more effective coupling through the developing network structure. Preliminary results showed that the gel peak could be intensified by use of a two ply specimen but there was then a reduction in the intensity of the vitrification peak. Furthermore, the higher rigidity of the two ply specimen exceeded the capability of the instrument (using the usual inertial mass) in the later stages of cure. The use of samples as wide as 1/4", the limit permitted by the usual grips, did not intensify the gel peak and in preliminary experiments appeared to cause double peaked behavior at vitrification. It will be seen in the results to be presented that single ply 2-1/2" long, 1/8" wide strips, which were used subsequently for the most part, proved satisfactory.

#### Prepreg I

The conditions recommended by the composite fabricator for the processing of I were 127°C/2 hrs. Isothermal runs were conducted over the temperature range 63 to 103°C and some measurements were also made very close to the ultimate glass transition temperature,  $T_{g\infty}$  (~160°C). Results are shown in Figure 2 for a cure at 69°C where gelation is indicated by the weak maximum which precedes the strong vitrification peak in the first time sweep (of 18.2 hr). Figure 3 shows a cure at 84°C where the strongest gel peak in this series was observed. At the two higher cure temperatures (95, 103°C) the gel peak was weaker but a gel time could be approximated by the point at which the damping curve turns downward to form the minimum which precedes vitrification

at these temperatures (Figure 4).

The gel peak in the damping curve can be related to a critical viscosity or to an isoviscous state which, because of the rapid increase in viscosity near the gel point, can be used as a measure of the time to gelation (1,3). If the mechanism of cure does not change with temperature then gelation will occur at a fixed chemical conversion (4). On this basis the gel times can be treated as being inversely proportional to the rates of reaction and can be plotted in a conventional Arrhenius form (Figure 5) to determine an apparent activation energy for the reactions leading to gelation. The value obtained is 24.4 Kcal/mole, which is substantially higher than the result reported earlier (19.7 Kcal/mole) for a sample of the same type of resin cured on glass braid (3).

The plots of the gelation and vitrification times versus isothermal temperature are shown in Figure 6 where the two curves appear to be parallel. At low temperatures the two curves must cross since the  $T_g$  of the ungelled glass can be determined on cooling. However, measurement of gelation times below the 63°C run were not made. At high isothermal temperatures the vitrification times must pass through a minimum before undergoing a rapid increase (1,2). The increase in vitrification time at temperatures approaching  $T_{g\infty}$  is due to the fact that only low concentrations of reactants remain as the glass transition of the reacting system reaches the higher temperatures of cure. For the same reason, at temperatures near  $T_{g\infty}$  there is difficulty in determining the vitrification point due to broadening of the peak maximum. This is shown by the results of a cure at 162°C (Figure 7).

It should be pointed out that the maximum  $T_g$  observed in the prior study of this resin was 147°C (3). The difference in  $T_g$  for the earlier resin sample and the current prepreg sample, as well as the difference in activation energies noted above, suggests the two resins, although bearing the same manufacturer's designation, may not be identical.

The results for the prepreg sample cured at 69°C and then cycled between -190 and 200°C are shown in Figure 8. A damping maximum is observed at 103°C on the initial heating cycle (-190 to 200°C) which corresponds to the  $T_g$  of the sample cured for a prolonged time at 69°C. Further curing occurs during heating to 200°C. On cooling from 200°C,  $T_g$  has moved upward to 156°C and the low temperature damping ( $T < T_g$ ) has also increased in temperature and intensity (cf. references 1 & 5). As shown in Figure 9 the  $T_g$  increases in direct proportion to the cure temperature with  $(T_g - T_{\text{cure}}) \approx 40^\circ\text{C}$  over the temperature range considered. After cycling to 200°C,  $T_g$  (200) is almost independent of the earlier cure history. However, as shown in Figure 7, vitrification can occur at temperatures higher than  $T_g$  (200). This behavior has been observed in other epoxy resins.

It is evident from Figures 1-4 and earlier work (3,6) that chemical reaction continues past the vitrification peak and the rigidity attains a limiting value at much longer times. This indicates that the reduction in molecular mobility required to quench chemical reactions occurs well beyond the measured transition. The results of Figure 9, which show that  $(T_g - T_{\text{cure}}) \approx 40^\circ\text{C}$ , imply that quenching of chemical reactions will occur at least 40° below  $T_g$ , which is into the glassy plateau region of the rigidity-temperature curve. If the operational

measurement of the glass transition and vitrification were to correspond to the quenching of chemical reactions then  $(T_g - T_{cure})$  would equal zero (1).

The magnitude determined for  $(T_g - T_{cure})$  will depend on a number of factors including any further cure which occurs during the scanning run itself (7). To examine this effect, heating scans were conducted at 1.5°C/min and 5°C/min on samples which had been thoroughly cured (> 20 hr) at 85°C. For the slow and fast heating rates the respective values of  $T_g$  were 123°C and 124°C which suggests that very little additional cure occurs during the first scan. After reaching 129°C the specimens in both cases were rapidly cooled and rescanned. The resulting  $T_g$  values of 145°C and 130°C, for the slow and fast heating rates respectively, indicate that additional cure occurs above the measured  $T_g$  even during the relatively brief time intervals involved in the heating scans.

#### Prepregs II and III

The recommended cure conditions for II and III are 177°C/2 hours. The isothermal cure study of these two samples was limited to a few illustrative experiments. At 148°C the isothermal cure curves for II and III (Figures 10, 11) are similar to those observed with I at lower temperature, although there is now some improvement in the definition of the gel peak. One cure was also carried out on III at 99°C, nearly 80°C below the recommended cure temperature with the results in Figure 12. The times to gel and vitrify are comparable to I run 50°C below its recommended cure temperature. This suggests a broad curing range for

III. In the DSC scan (30° - 260°C) some exothermic reaction can also be observed at temperatures as low as 100°C.

#### Temperature-Programmed Cure

The temperature scan on the uncured prepreg I (-35 to 200°C, 0.05°C/min) in Figure 13 shows in succession the  $T_g$  of the uncured resin (6°C), the " $T_{\ell\ell}$ " relaxation (1,8), a small but distinct gel peak (82°C), a marked vitrification peak (89°C) and a somewhat broad devitrification process (159°C) with corresponding changes in rigidity. The results for cycling an uncured sample of prepreg III between -190 and 200°C are shown in Figure 14. A  $T < T_g$  relaxation, the  $T_g$  of the uncured resin (20°C), a  $T_{\ell\ell}$  relaxation, gel (165°C) and vitrification (195°C) temperatures are evident but the final temperature of the experiment was too low to display the devitrification process. The second cycle displays the marked high temperature shift, and the broadening and intensification of the low temperature damping process which occurs with cure in this resin, in contrast to smaller changes in I (Figure 15). This intense, broad damping peak with a maximum somewhat below room temperature in III (and II) presumably would contribute to improved impact performance of the cured composite.

Temperature scans at different heating rates have also been performed on the various prepreg samples. Figure 16 for II and III shows that there is a close correspondence between the temperatures of vitrification ( $T_{vit}$ ) and the temperatures of gelation ( $T_{gel}$ ) for these two samples but about a 10°C difference in the initial  $T_g$  values. This suggests that the resins used in the two prepregs are substantially the

same and differ primarily in the degree of advancement. The  $T_g$  values of the three uncured preregs determined by DSC show good correlation with the TBA results but occur about 10°C lower in temperature.

The results obtained from the scans at different heating rates can be also used to construct a time-temperature cure-type of state diagram similar to that defined by isothermal experiments (1). The general behavior expected is illustrated by Figure 17 taken from reference 5. In the TBA scans at very slow heating rates (curve e) gelation, vitrification and devitrification will be observed (see also Figure 13). At high heating rates vitrification does not occur since the sample temperature is advancing faster than the degree of cure. However, the transformation to the rubbery state is observed (e.g., curve b, Figure 17), gelation will occur at a fixed conversion independent of the sample temperature. It appears that at the heating rates commonly used in DSC scans ( $> 1^\circ\text{C}/\text{min}$ ) the cure behavior will not be affected by vitrification. In principle, by combining DSC and TBA scans at lower heating rates it should be possible to estimate the conversion at gelation but since the DSC response decreases with decreasing heating rate this might not be feasible.

#### CONCLUSIONS

This study has shown that substantially all of the information concerning the curing behavior of epoxy resins which has been obtained for resin samples on glass braid can also be obtained working directly with prepreg materials. Although the gel peak is weaker, this event can be determined with sufficient accuracy to construct an Arrhenius

plot over a wide range of temperatures for the determination of an activation energy. Scans on the uncured resin can give the  $T_g$  of the prepreg which is a sensitive measure of the extent of B staging as well as any storage aging suffered by the material (9). In addition the isothermal cure on the same sample, or a temperature scan, can provide a measure of the other curing parameters.

#### ACKNOWLEDGMENTS

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## FIGURE CAPTIONS

- Fig. 1. Isothermal cure, Prepreg I, 90°C.
- Fig. 2. Isothermal cure, Prepreg I, 69°C.
- Fig. 3. Isothermal cure, Prepreg I, 84°C.
- Fig. 4. Isothermal cure, Prepreg I, 103°C.
- Fig. 5. Arrhenius plot, Prepreg I.
- Fig. 6. Gelation and vitrification times versus temperature, Prepreg I.
- Fig. 7. Isothermal cure, Prepreg I, 162°C.
- Fig. 8. Scan, Prepreg I from isothermal cure at 69°C/62 hr; plot, 69 → -190 → 200 → -190°C, 1.5°C/min.
- Fig. 9.  $T_g$  and  $T_g(200)$ , Prepreg I.
- Fig. 10. Isothermal cure, Prepreg II, 148°C.
- Fig. 11. Isothermal cure, Prepreg III, 148°C.
- Fig. 12. Isothermal cure, Prepreg III, 99°C.
- Fig. 13. Scan, uncured Prepreg I: 20 → -35°C/1.0°C/min; plot, -35 → 200°C, 0.05°C/min.
- Fig. 14. Scan, uncured Prepreg III; plot, 20 → -190 → 200 → -190 → 200°C; 1.5°C/min.
- Fig. 15. Scan, uncured Prepreg I: plot 20 → -190 → 200 → -190 → 250 → -190°C; 1.5°C/min.
- Fig. 16.  $T_{vit}$ ,  $T_{gel}$ ,  $T_g$  versus heating rate for Prepreg II and Prepreg III.
- Fig. 17. Time-temperature cure state diagram from experiments at different heating rates (5).  
a) 5°C/min, b) 1.5°C/min, c) 0.75°C/min, d) 0.25°C/min, e) 0.05°C/min.



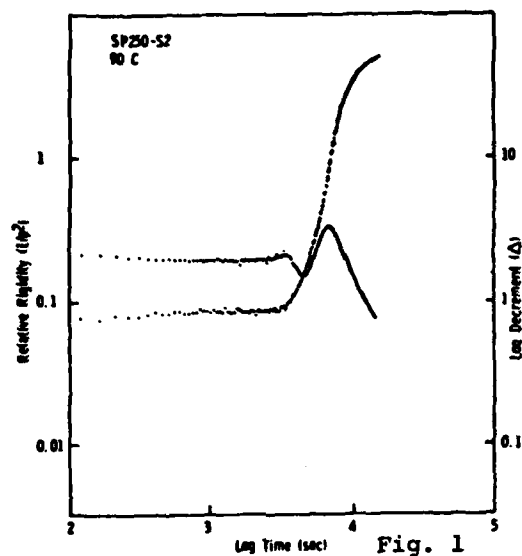


Fig. 1

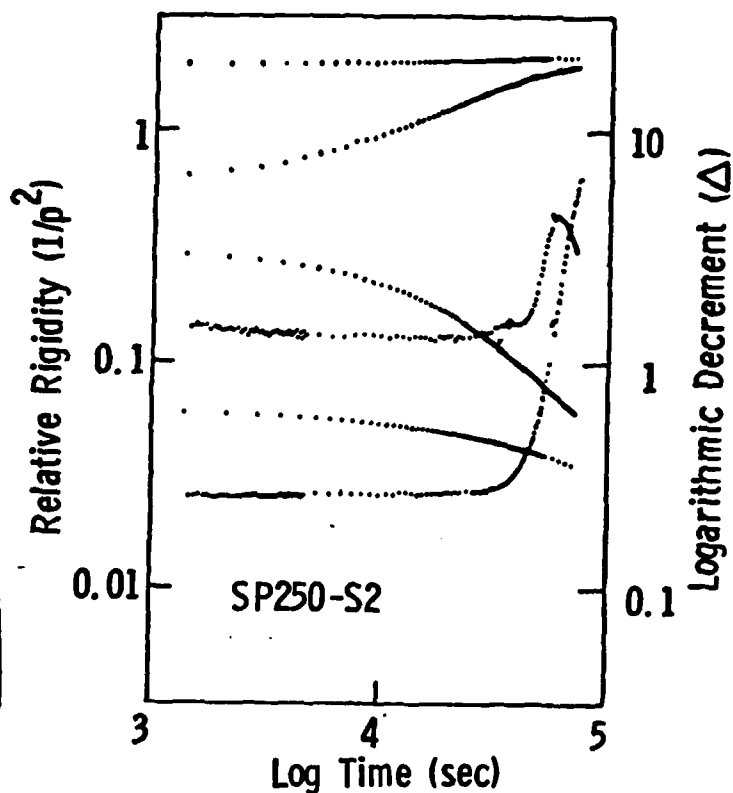


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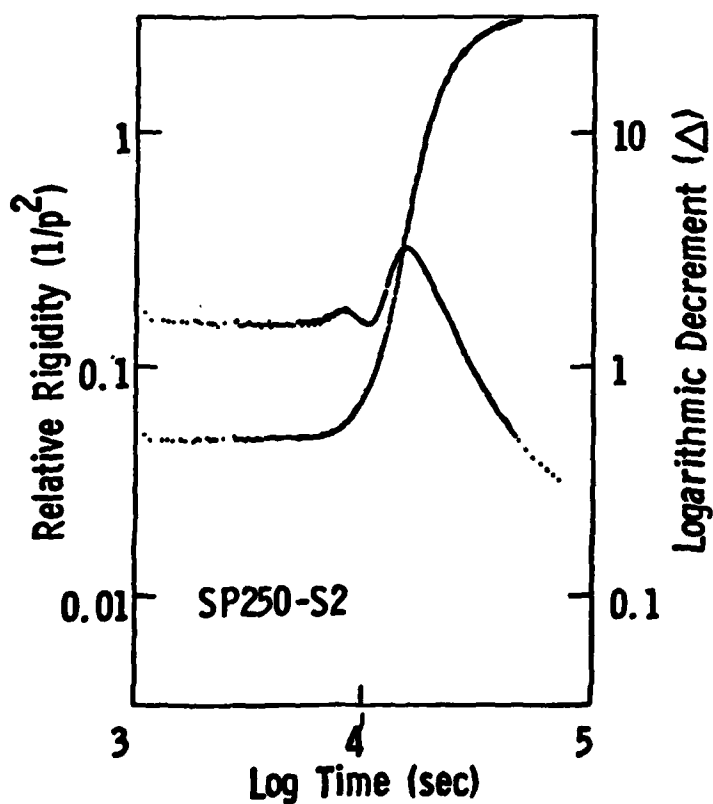


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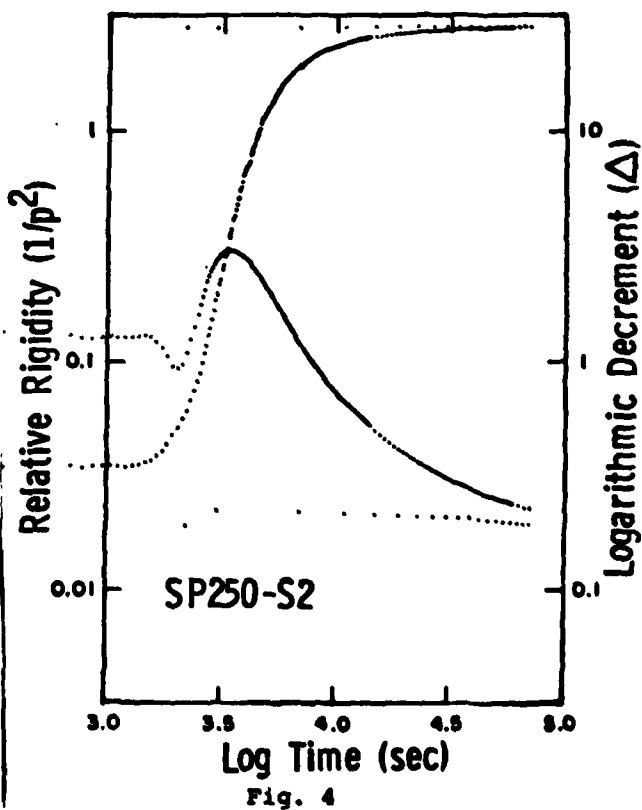


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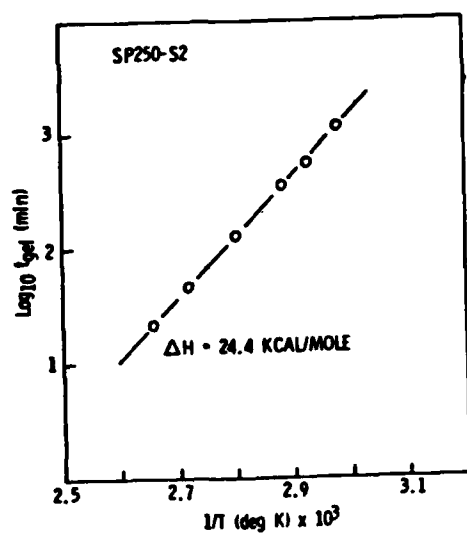


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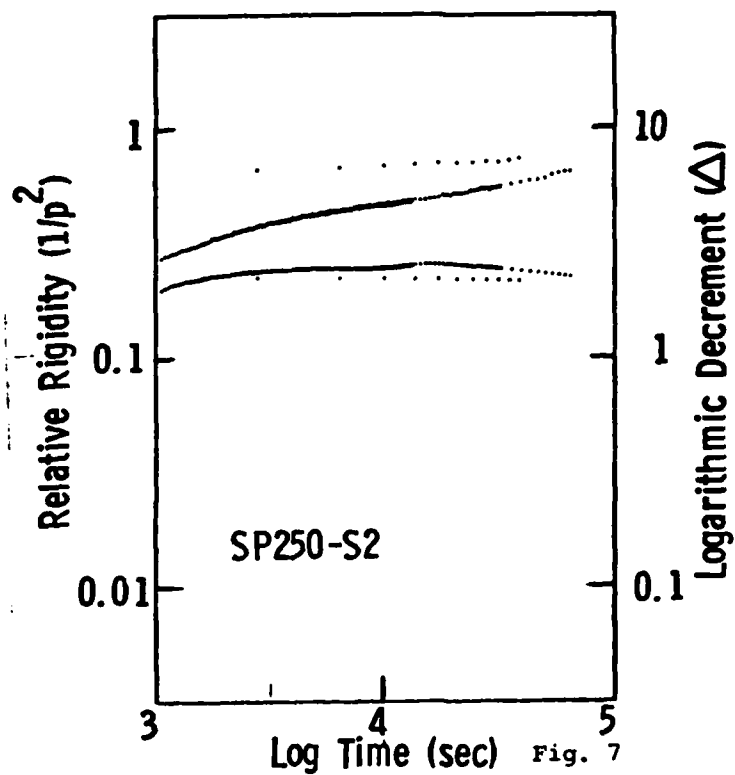


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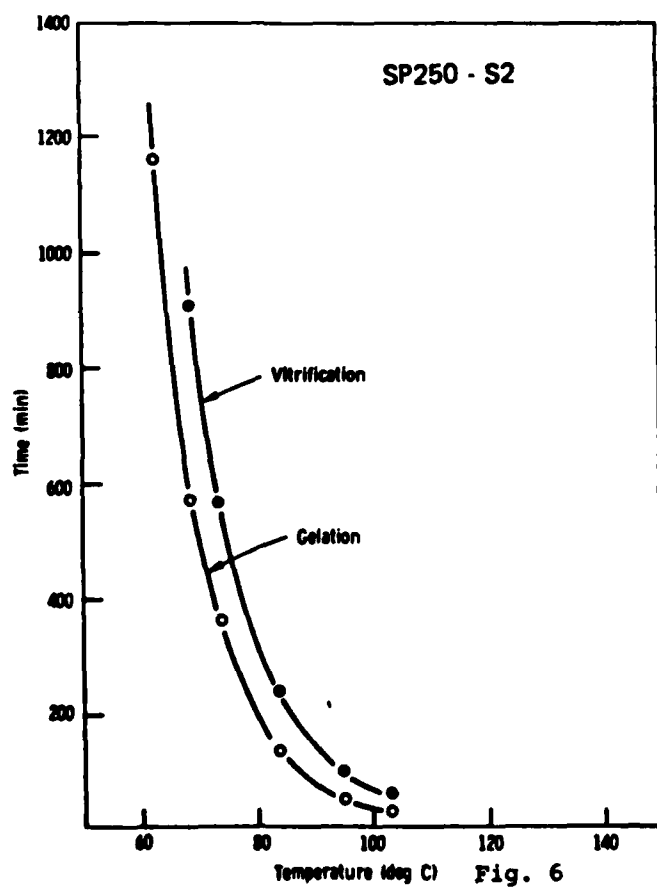


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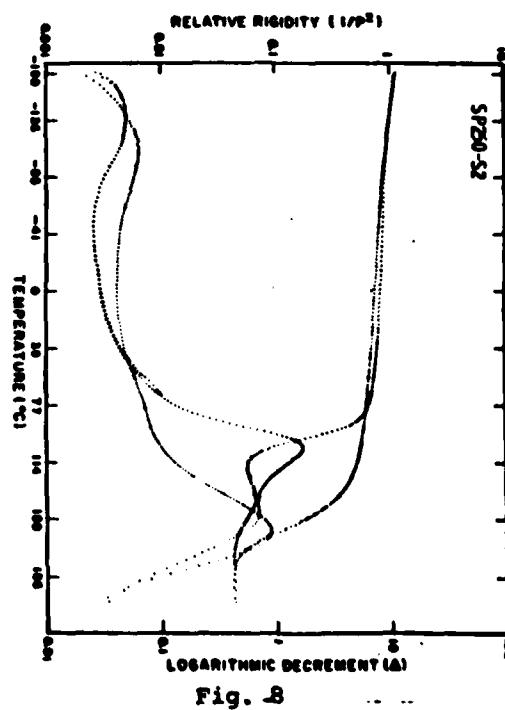
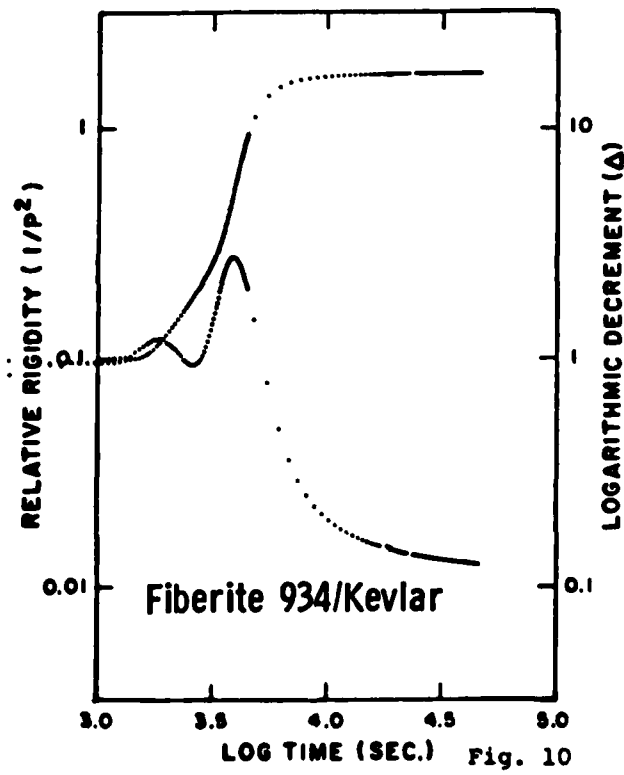
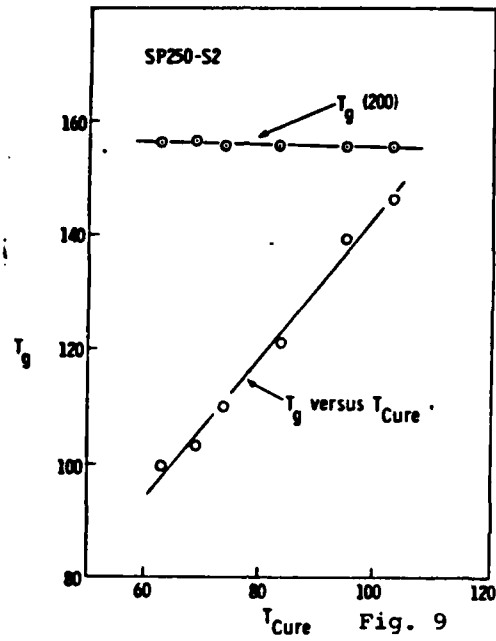
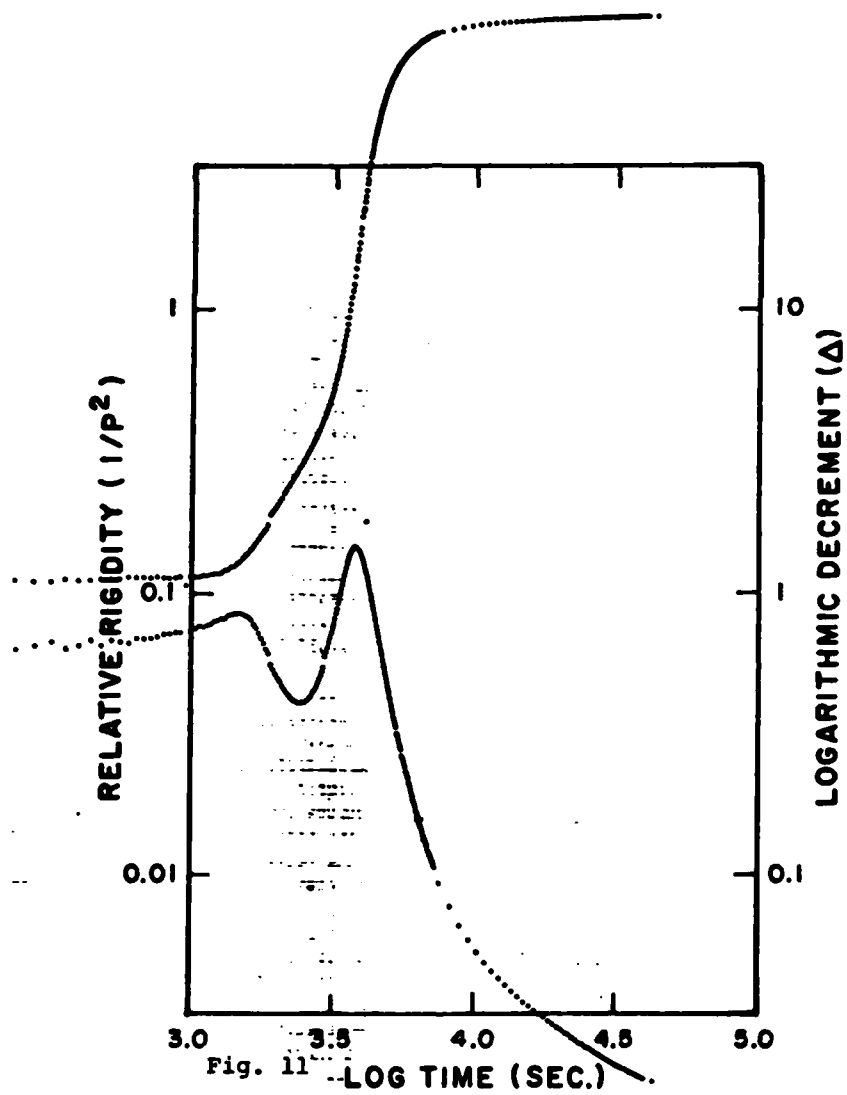
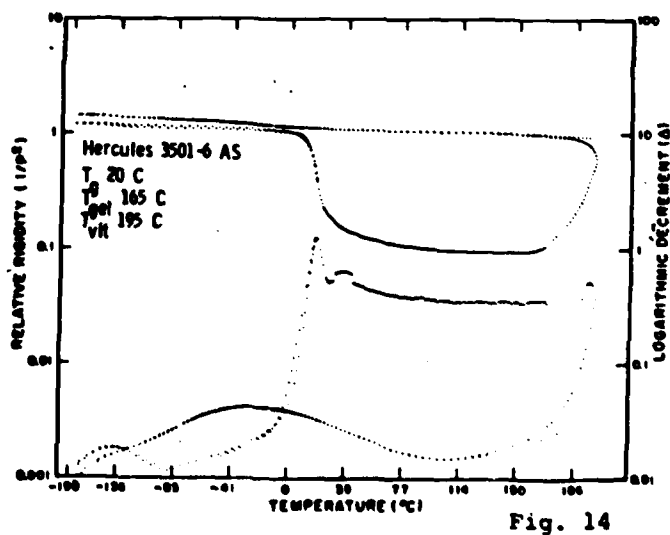
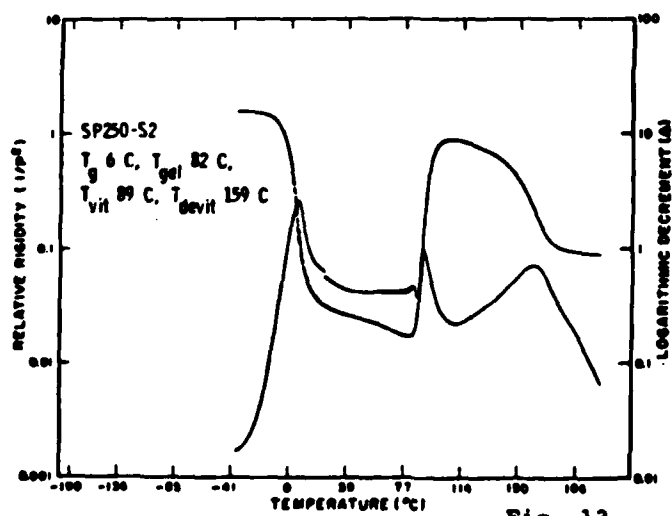
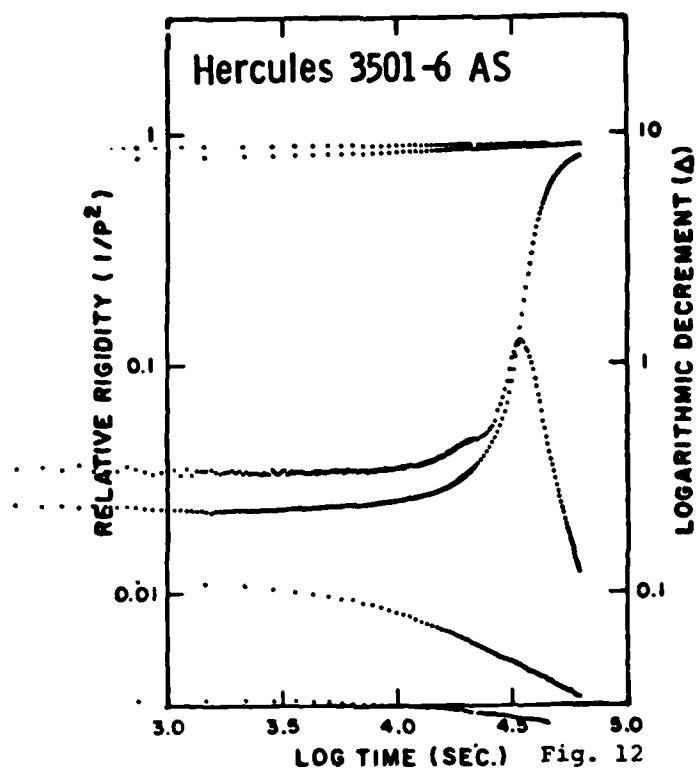


Fig. 8







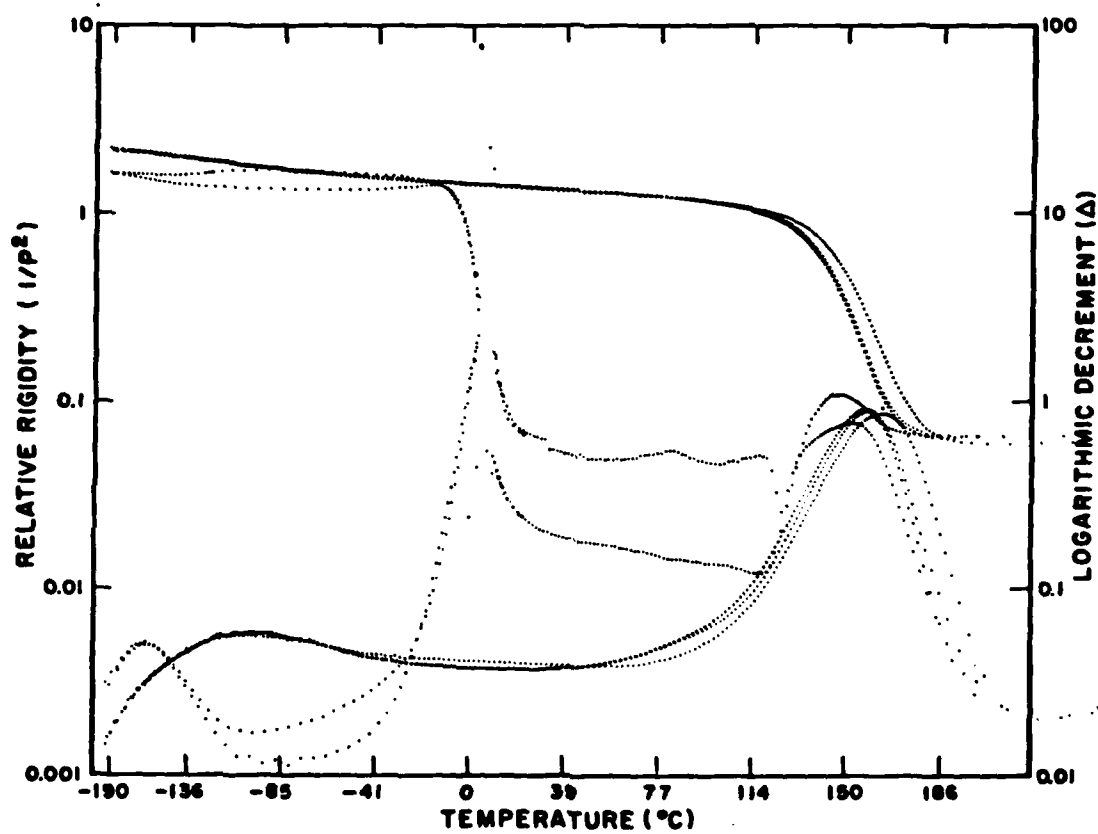


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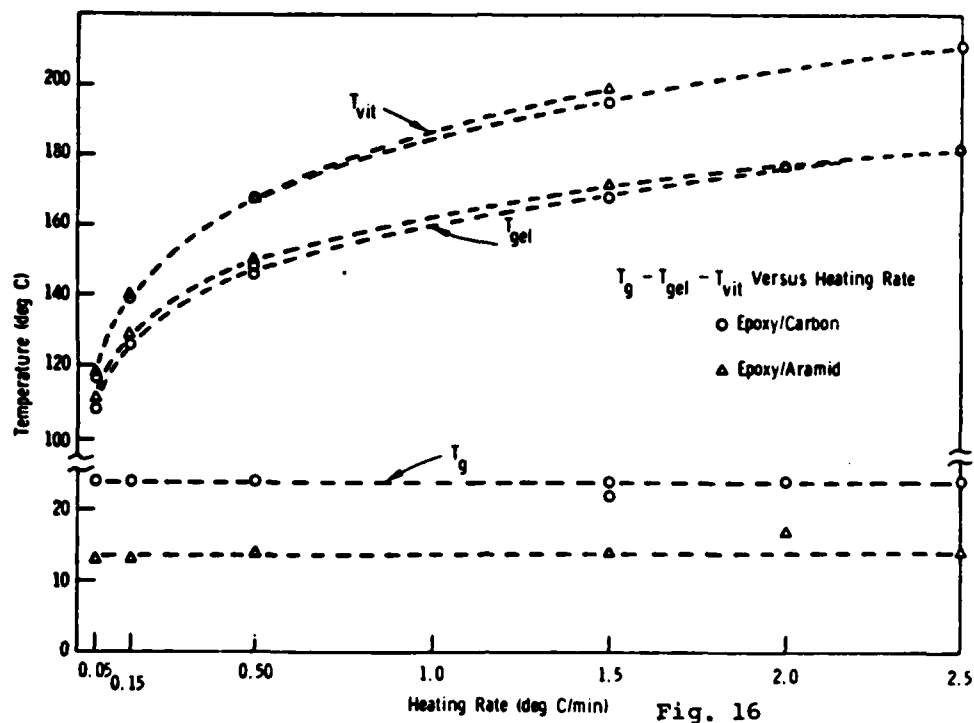


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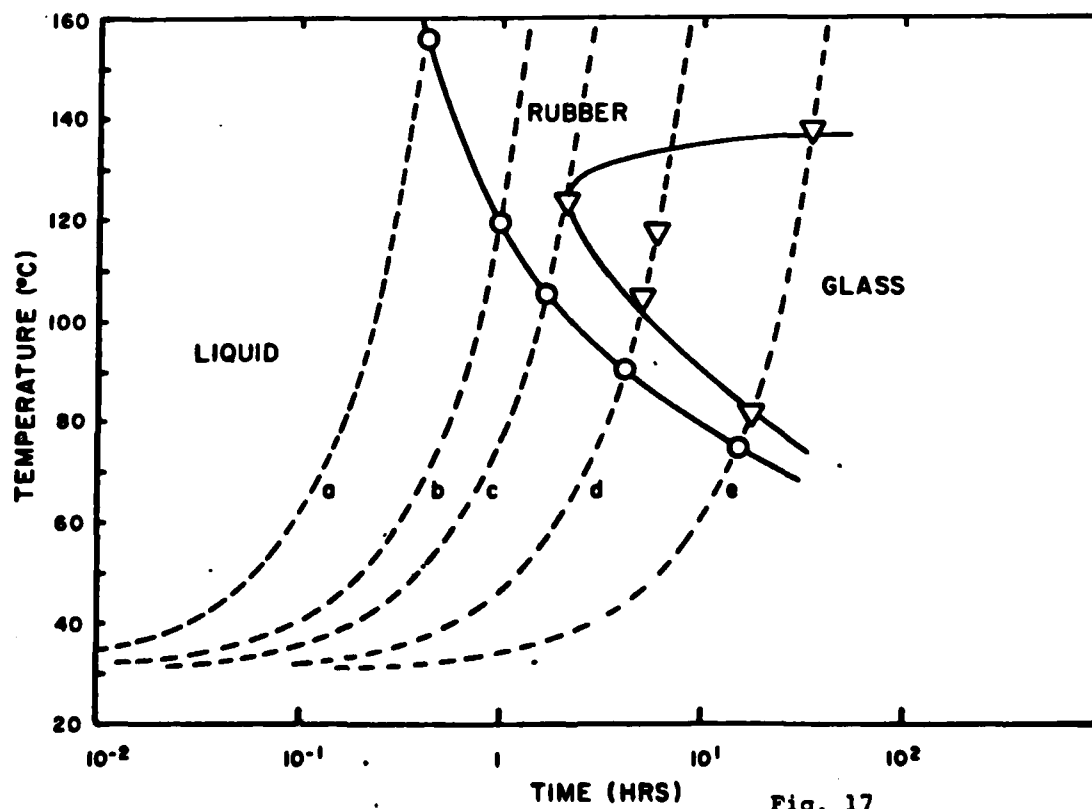


Fig. 17

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